BEYER

The Fusion of Clays
Shales etc. by Means of
Sodium Peroxide

Chemical Engineering B. S.

1907.



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## THE FUSION OF CLAYS, SHALES, ETC., BY MEANS OF SODIUM PEROXIDE

 $\mathbf{B}\mathbf{Y}$ 

GEORGE FREDERICK BEYER

### THESIS

FOR

#### DEGREE OF BACHELOR OF SCIENCE

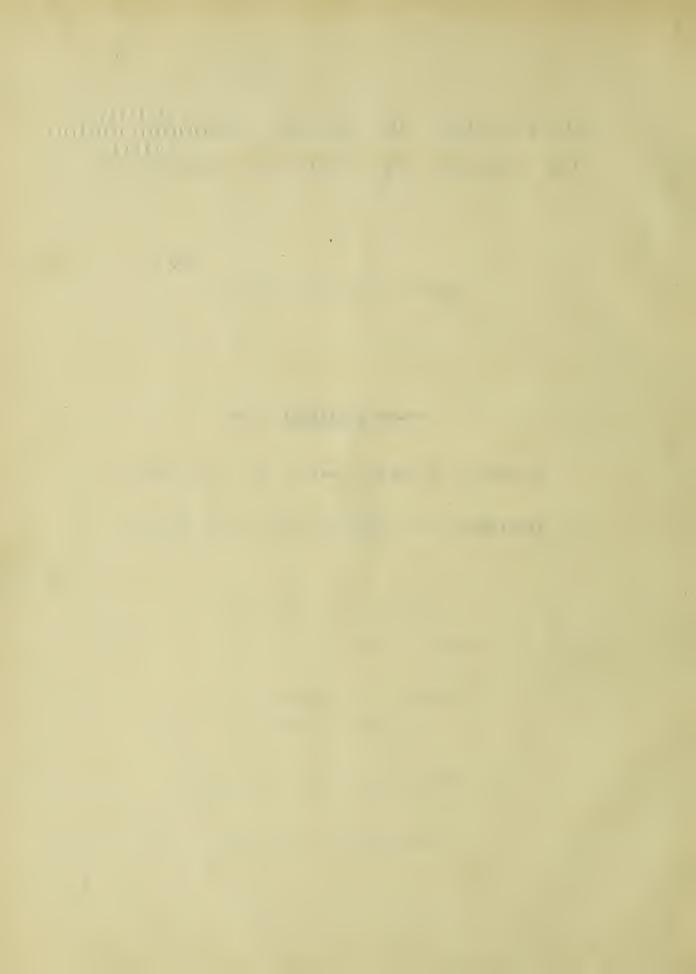
IN

CHEMICAL ENGINEERING

COLLEGE OF SCIENCE

UNIVERSITY OF ILLINOIS

PRESENTED JUNE, 1907



#### UNIVERSITY OF ILLINOIS

May 31 1907.

THIS IS TO CERTIFY THAT THE THESIS PREPARED UNDER MY SUPERVISION BY

George Frederick Beyer

The Fusion of Clays, Shales, Etc., by Means of

Sodium Peroxide.

IS APPROVED BY ME AS FULFILLING THIS PART OF THE REQUIREMENTS FOR THE

DEGREE OF

Bachelor of Science

in Chemical Engineering.

HEAD OF DEPARTMENT OF Chemistry.

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The Real Problems of

#### INTRODUCTION.

For analytical work on silicates of all kinds the most important condition is the obtaining of a perfect fusion, and that with the greatest possible rapidity. Up to the present time probably the only fusion mixtures that have proven a success for both analytical and practicable purposes are sodium carbonate and potassium carbonate.

The above fusion mixtures are all well and good as far as getting the substances into solution, but when a certain degree of rapidity is required, great difficulty is experienced. The time element is not the only thing required, for, when sodium carbonate is used as the fusion of a mineral, a great amount of heat is necessary for a considerable length of time. Thus, if some method could be devised or discovered in which the time element and the application of external heat could be eliminated and still give a perfect fusion, it would prove of considerable help and advantage to both the practicable and the analytical chemist, and the question of fusion mixtures and slags would be solved.

It has been known for quite a long time that when sodium peroxide was used in conjunction with sodium carbonate and other substances, a considerable amount of heat was generated and that the reaction was rather violent. When sodium peroxide was used alone in an open crucible the reaction was too violent, and consequently, for a large number of substances



it could not be used advantageously. Its great oxidizing power was also known well as its power of generating heat when reacting with other substances. If these properties could be made use of in some way it is possible and quite probable that a fusion could be obtained that is suitable for practically all analytical purposes.

It is probably well to enumerate here some of the advantages and disadvantages of the methods that are now generally employed for the fusion of clays, shales and other substances of a similar nature.

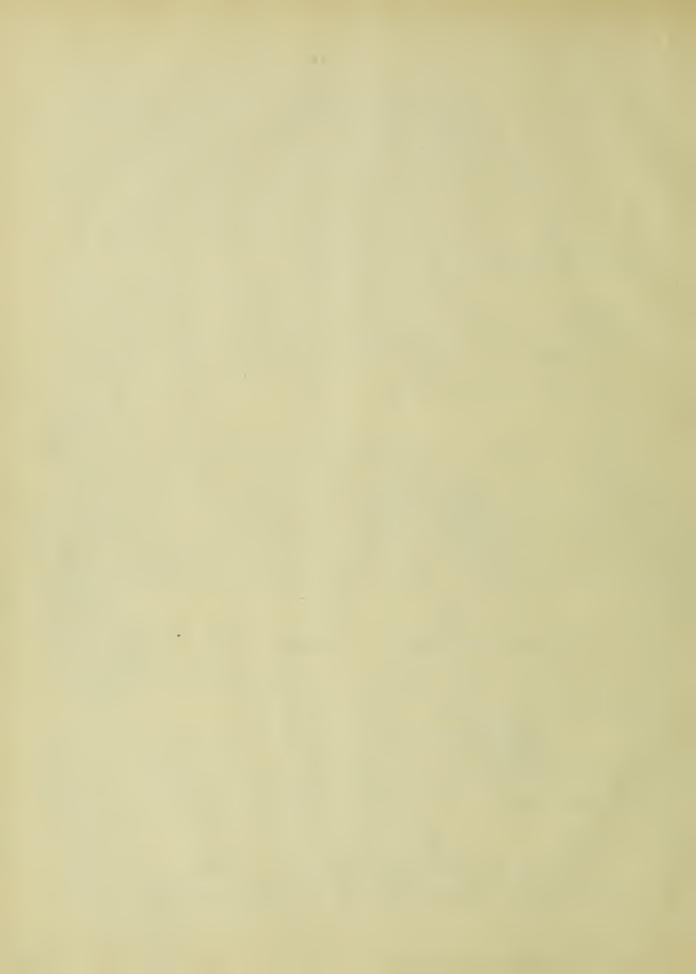
Methods of Decompositions of Silicates.

The methods that are generally in use now for the decomposition of silicates are as follows:

- (a) Solution in hydrochloric or other acids. (1)
- (b) Solution in HCl in a closed tube using benzene on the outside.
- (c) Berzelius' Method. Solution in HFl and  $\rm H_2SO_4$ . (2) The chief advantage of this method is, that no other mineral is introduced into the solution.

The disadvantages are, that it is almost impossible to get the silicate decomposed and in order to completely get it into solution it takes many hours of digesting with the acids.

(d) Fusion with sodium carbonate, or better, a 1 to 1 mixture of sodium carbonate and potassium carbonate. (13)



The chief advantage over the other methods mentioned is that the silicate is much more easily decomposed and in about 30 minutes a complete fusion is obtained.

The main disadvantages lie in the fact that sodium carbonate and potassium carbonate are liable to contain impurities, as they are scarcely ever obtained chemically pure. The heat of an ordinary Bunsen burner is not sufficiently high to bring about a fusion. As a matter of fact a half hour means considerable waste of time to many Chemists, especially when many Feldspar analyses have to be made. Therefore, a complete decomposition in less time, other things being equal, would mean a great economy in time.

(e) Boric Acid Method (Januarch and Heidenreich) (3)

The thing mainly in favor of this method is that borax has the property of dissolving oxides of minerals and forms easily fusible borates. Boric acid can be obtained very pure.

The disadvantages are that when boric acid is heated it breaks up into  $B_2 O_3$ , which is very volatile, and gets all over the laboratory and makes it almost impossible to work with it. A very high temperature is also needed for the complete decomposition of the mineral.

(f) Lead Oxide Method of Jannasch. (4)

The method consists of heating the finely ground substance in a platinum crucible or dish with lead carbonate for 15 to 20 minutes over a low flame by means of which the greater part of the carbon dioxide is driven off. Then the



contents of the crucible are more strongly heated until the substance is completely decomposed.

The chief advantage of this method is that all the elements contained in the substance may be determined from a single fusion.

The disadvantages of this method are about the same as in all the others, viz., the time it takes to fuse the substance is entirely too long, and the danger in use of the expensive platinum dish.

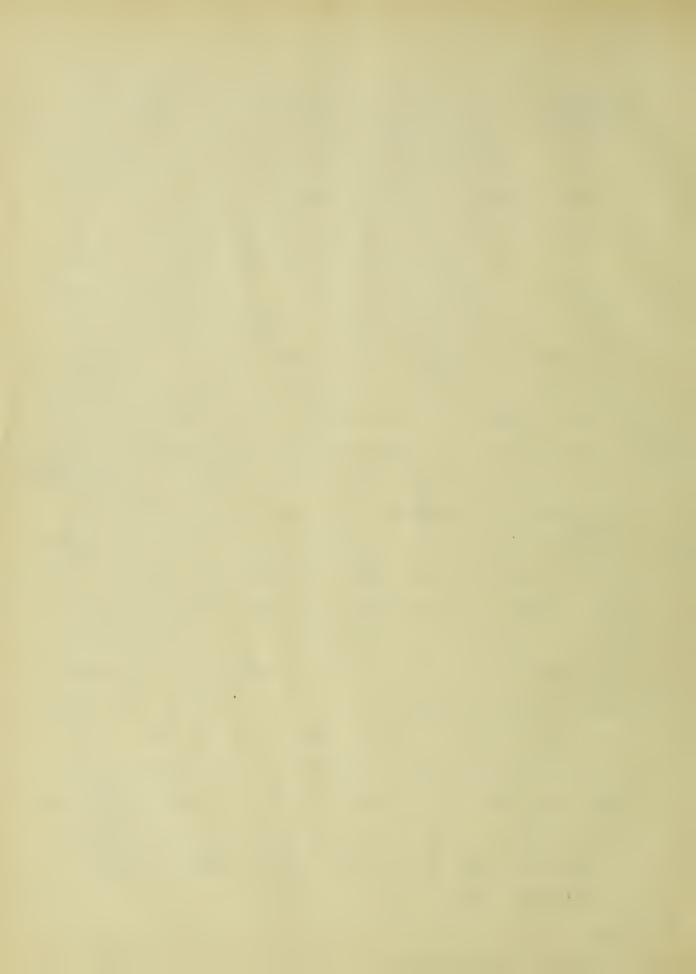
Use of Sodium Peroxide for the Determination of S
in Coke and Coal. (5)

Statements have been made at various times, and by as many chemists that sodium peroxide could not be used in the determination of sulphur in coal and coke, because its action was too violent, which caused spurting and consequently a loss of the material. C. Glaser disputes the above statement and says that the reaction is not too violent if treated properly. His method, briefly, is to treat the coal or coke in a large nickel dish with about 4 times its weight of sodium carbonate, and upon that a piece of sodium hydroxide. The dish is now slowly moved over a low flame until a half fused mass is obtained, upon which small quantities of dry sodium peroxide are dusted at different intervals, waiting each time until all the reaction is over. This is continued until all the carbon is burned away, when the mass, if necessary, is heated to a



perfect fusion. If the above directions are followed out closely and carefully there will be no loss by explosion or spurting. The sulphur is then determined in the ordinary way. The time required to completely fuse the coal by this method is about 15 minutes, to fuse coke about 10 minutes.

It has also been said that chrome ores can not be fused by the use of sodium peroxide. It can be readily seen that by any of the above methods it takes considerable time and skill in manipulation and the greatest of care to make a determination of sulphur in the aforesaid substances, the liability of losing the whole substance at any moment being great. A method has recently been devised for decomposing coal and coke by the use of sodium peroxide which obviates all the above elements of disadvantages. The method is one in which the material for analysis is placed in a nickel-steel bomb treated with a certain amount of sodium peroxide and a mixture of potassium chlorate and benzoic acid, the whole of which is mixed thoroughly and then ignited by passing an electric current through an iron wire of very high resistance and small diameter. The mass fuses instantly, and is left in such a condition that it can be readily removed from the bomb. time to perform the whole experiment is less that the time it takes to perform the first half of the above method of Glaser's. This clearly, and without a doubt, shows the great advantage of using the sodium peroxide in a closed chamber, namely, a nickel-steel bomb.



"Sodium Peroxide is an excellent substitute for the ordinary fusion mixtures", says W. Hempel (6), "and is well adapted for the dry oxidation of mineral substances. Chromium and Manganese ores, for example, when heated with twice their weight of peroxide, yield almost immediately a yellow or green melt, as the case may be. Wolfram, fused with four times its weight of the reagent, is converted in a few minutes into sodium tungsten. Titaniferous iron ores are also rapidly and completely decomposed. Fusion with peroxide may be advantageously employed in the case of zinc blende and galena, when a full analysis is required, since any iron remains in the insoluble residue, while the zinc and lead pass into the alkaline solution."

substances, both quantitatively and qualitatively, sodium peroxide may be used quite advantageously. The ignition of organic substances with the reagent may be employed for the rapid and simultaneous qualitative detection of the halogens,
phosphorous, arsenic, and sulphur. (7) A mixture of sodium
peroxide with 1/25 part of an organic, non-hygroscopic substance such as naphthalene, or cinnamic acid is prepared and
used in this analysis. The substances to be analyzed are
mixed thoroughly and placed in an iron crucible, then heated
until the reaction occurs. When a quantitative analysis of
the halogens is desired, the substance is mixed with about
20 times its weight of sodium peroxide, ignited in a covered



iron crucible by passing a hot wire through a hole in the cover. The product is then dissolved in water, acidified with nitric acid and precipitated with silver nitrate. Nitrogen may also be determined in organic substances, such as flour, by the use of the peroxide. (12) After the flour has been dried at 100° it is mixed in a steel cylinder with an excess of the dry reagent, to which a mixture of potassium persulphate and tartaric acid had been added. The cylinder is kept cool in water, and when the vigorous oxidation which has been started by means of a red hot iron wire, is complete, the whole is dissolved in water and then the nitrogen is determined in the ordinary way.

The Action of Fused Sodium Peroxide on Metals. (8)

Nickelo Nickelic oxide Ni<sub>3</sub>0<sub>4</sub> was prepared by heating metallic Ni in a Ni crucible with sodium peroxide at a cherry red heat for about 30 minutes. It is a black lusterless amorphous powder which is soluble in hydrochloric, sulphuric and nitric acids.

Iron is rapidly attacked by fused sodium peroxide, especially if it be heated to bright redness. An ordinary sheet iron crucible will rarely withstand more than one or two fusions with perforations. Clippings of iron put into the crucible will protect it to some extent and at the same time hasten the action of the sodium peroxide. If the fusion



is kept at bright redness for 30 minutes the reaction is usually complete, but at dull redness some of the sodium peroxide will remain undecomposed for 3 1/2 hours or longer.

The compound obtained after fusion is  $\mathrm{Fe}_2\mathrm{O}_3\mathrm{H}_2\mathrm{O},$  and is magnetic.

Fused sodium peroxide attacks gold very rapidly and leaves it in a spongy state. The gold seems to be oxidized, but the oxide formed decomposes at the temperature of the fusion of the sodium peroxide and does not re-form on cooling, therefore the gold remains in the form of sponge. The compound formed is  $\mathrm{Au_2O_3}$  which gives off some 0 at 110°C, and at 160°C. Au0 remains which in turn is oxidized at 250°C, leaving mettalic gold.

Silver is also attacked by sodium peroxide. In fact, so rapidly is it attacked that after 3 or 4 fusions of sodium peroxide in a silver crucible it is perforated even when scraps of silver are added. When the fusion is conducted at a dull red heat, the action ceases in about 20 or 30 minutes, and the fused mass will be filled with needle-shaped crystals. Platinum is also rapidly attacked by fused sodium peroxide and if in a spongy or finely divided state, and ultimately mixed with it, the platinum burns almost like tinder when heated to bright redness. If the combustion be started in a portion of the mixture, it is transmitted rapidly throughout the mass, which becomes heated to bright redness. A platinum crucible withstands very few fusions of sodium peroxide.



Here are a few reactions showing the reaction of sodium peroxide on Pt.

2 Pt 3 Na<sub>2</sub>0<sub>2</sub> = Pt<sub>2</sub>0<sub>3</sub>, 2 Na<sub>2</sub>0<sub>2</sub> Na<sub>2</sub>0<sub>2</sub>. Pt<sub>2</sub>0<sub>3</sub>, 2Na<sub>2</sub>0<sub>2</sub> 4Hc<sub>2</sub>H<sub>3</sub>0<sub>2</sub> = Pt<sub>2</sub>0<sub>3</sub>, 2H<sub>2</sub>0 4 Nac<sub>2</sub>H<sub>3</sub>0<sub>2</sub>. Pt<sub>2</sub>0<sub>3</sub>, 2Na<sub>2</sub>0<sub>2</sub> 4H<sub>2</sub>0 = Pt<sub>2</sub>0<sub>3</sub>, 2H<sub>2</sub>0 4NaOH.

Sodium peroxide may also be used quantitatively in the separation of Sb, Sn and As (9). The method is, in a few words, as follows. The mixed sulphides are stirred with a small amount of water and sodium peroxide is added by degrees until a small portion no longer gives a colored precipitate on the addition of diluted  $\mathrm{H_2SO_4}$ . The whole is then transferred to a silver crucible, evaporated to dryness, and kept in a state of fusion for some time. The process from then on is similar to the ordinary one of separation.

When titanium is present in any ore it may be determined gravimetrically by the use of sodium peroxide. (10)

The finely ground ore is placed into either a silver, nickel or iron crucible and treated with sodium peroxide and some sodium carbonate. The amount of sodium peroxide is always greater than the amount of sodium carbonate. The above substances, after being thoroughly mixed, are heated at a low temperature for about 20 minutes. The rest of the determination is carried out from this stage according to any of the standard methods.

The silicon in rich Ferro-silicons can be rapidly and accurately determined by means of peroxide of sodium. (11)



If the finely ground ore is intimately mixed with about 20 times its weight of a mixture of sodium carbonate and potassium carbonate and I gram of sodium peroxide, then heated slow-ly over a low flame, the reaction will be quiet, and the decomposition complete. The fused mass is then analyzed for silicon in the ordinary way.

#### Object.

The object of the experiments performed was to find some mixture of accelerators with sodium peroxide that, on being ignited in a closed chamber, would completely decompose any clay, shale or mineral in the least possible time.

In the course of the experimenting it was found that certain mixtures would give combustions that were entirely satisfactory, with the one objection, that the fused mass boiled to the top of the bomb, thus making the fusion very difficult to remove. It was also noted that it was absolutely necessary to put on a false top while mixing the ingredients, because particles of the clay, or substance to be fused would invariably adhere to the prongs and bottom side of the regualr top; consequently preventing them from coming in contact with the fusion mixture.



#### Experimental.

A nickel-steel cylinder or bomb was used to carry out the following experiments. This kind of a bomb has been used but very little, especially the one in which the substance is ignited by passing an electric current through an iron wire.

The purpose of the first experiment was to find some mixture of an oxidixing and some other organic, non-hygroscopic substance that would ignite readily, rapidly and generate a considerable quantity of heat. For that purpose the following experiments were carried out.

- 0.5 gm. clay.
- 1.0 gm. B(OH) KNO (6 to 1).
- 0.1 gm. Mg.
- 10.0 gm. Na202.

When the above substances were mixed in the bomb, it was impossible to ignite the mixture.

The same substances were used in a second trial with the addition of 0.2 gm. more of magnesium and 0.1 gm.of starch, but still the mixture would not ignite when the current was passed through the small iron wire.

- 1.0 gm. Boro nitrate mixture.
- 0.5 gm. Zinc dust.
- 0.5 gm. Clay.
- 10.0 gm. Sodium peroxide.



Still no combustion was obtained.

The same mixture, with the exception of the clay, was again tried, but the substances would not ignite.

As a result of these experiments it was concluded that some substance must be present that would furnish carbon for the combustion. So experiments were performed in which carbon was used in the form of coal and organic salts.

- 1.0 gm. Boro nitrate mixture.
- 0.5 gm. Zinc dust.
- 0.5 gm. Coal.
- 10.0 gm. Sodium peroxide.

This mixture ignited very slowly, and consequently gave a very poor combustion, leaving a considerable quantity of the coal unused. There was also a comparatively large quantity of gas generated during the reaction.

- 1.0 gm. Boro nitrate.
- 1.0 gm. Zinc dust.
- 0.5 gm. Starch.
- 10.0 gm. Sodium peroxide.

Ignited, but gave a rather poor combustion, however, less carbon remained than when coal was used. The combustion took place rather slowly, and a considerable amount of gas was formed.

Now that a mixture was found that would ignite, some



clay was added to see whetheror not it would be decomposed by the reaction.

- 1.0 gm. Boro nitrate.
- 1.0 gm. Zinc dust.
- 0.5 gm. Starch.
- 0.5 gm. Clay.
- 10.0 gm. Sodium peroxide.

The above mixture ignited very slowly and gave a poor combustion. There was some of the sodium peroxide left, not all being used. Most of the clay remained unattacked. Considerable gas was also formed.

- 1.0 gm. Boro nitrate.
- 1.0 gm. Zinc dust.
- 0.5 gm. Starch.
- 0.5 gm. Clay.
- 0.2 gm. Magnesium.
- 10.0 gm. Sodium peroxide.

This mixture ignited quite a little faster than the previous one and gave a fair combustion. Only an ordinary omount of gas was generated and most of the clay was fused; only the coarser particles remaining. The zinc dust seemed to retard the reaction and also another element would be added for determination when making a complete analysis. In using Mg, this would also be a disadvantage so it was decided to drop both out of the mixtures.



- 1.0 gm. Boro nitrate.
- 0.5 gm. Starch.
- 0.5 gm. Clay.
- 0.3 gm. Mg.
- 10.0 gm. Sodium peroxide.

Ignited fairly well, but gave a poor combustion, most of the clay remaining. The increase in magnesium seems to increase the amount of gas, and therefore, increases the pressure, which if it becomes too great, will rupture the bomb.

The Boro nitrate mixture was now dropped, but the magnesium was still used.

- 10.0 gm. Sodium peroxide.
  - 0.5 gm. Starch.
  - 0.5 gm. Clay.
  - 0.1 gm. Mg.

Ignited, and gave a comparatively good combustion, only the coarser particles remaining. There was just a little gas evolved. It seemed at this point, that if the clay were in the form of an impalpable powder it could be fused with the above mixture. This was tried and a determination of silica made. It may be said here, that it was concluded that if the silica, iron and aluminum could be determined and the results checked with those of standard methods, that would be sufficient to warrant the use of the method in the future.



For this purpose the following mixture was weighed out.

- 10.0 gm. Sodium peroxide.
  - 0.5 gm. Starch.
  - 0.5 gm. Clay (accurately).
  - 0.1 gm. Mg.

The clay was ground to a very fine powder and when ignited with the above mixture a very good combustion was obtained. Practically all the clay went into solution. The silica was determined by the ordinary method, but duplicates failed to check, and both results were low as compared with those of the standard methods. From this it was concluded that the combustion was incomplete, and as a result another mixture was sought for.

- 10.0 gm. Sodium peroxide.
  - 0.5 gm. Clay.
  - 0.3 gm. Sugar.

Combustion was only fairly good and a considerable amount of gas was evolved.

- 10.0 gm. Sodium peroxide.
- 0.5 gm. Clay.
- 0.4 gm. Sugar.

It was thought that by increasing the amount of sugar the amount of heat would be increased and thus make the

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combustion more complete. This, however, was not the case, for the above mixture, when fused, has not much better than the previous ones. Next the amount of clay was reduced.

- 10.0 gm. Sodium peroxide.
  - 0.3 gm. Clay.
  - 0.4 gm. Sugar.

The combustion was about the same as that above.

A mixture was now sought for that had a high calorific value, and for this  $B(OH)_{\odot}$  and  $NH_{A}Cl$  were used.

- 0.5 gm. Boric acid.
- 0.5 gm. Clay.
- 0.3 gm. Sugar.
- 10.0 gm. Sodium peroxide.
  - 0.2 gm. Ammonium chloride.

The mixture ignited very slowly and the combustion was not nearly so complete as the previous one.

- 0.5 gm. Clay.
- 0.3 gm. Sugar.
- 10.0 gm. Sodium peroxide.
  - 0.5 gm. Boro-nitro-Mg mixture.

I was unable to ignite the above mixture after attempting three times. Different mixtures were now tried.



- 0.5 gm. Clay.
- 1.0 gm. Ammonium oxelate.
- 7.0 gm. Sodium peroxide.

This gave only a fair combustion, ignited rather slowly, and a considerable amount of gas was generated during the reaction.

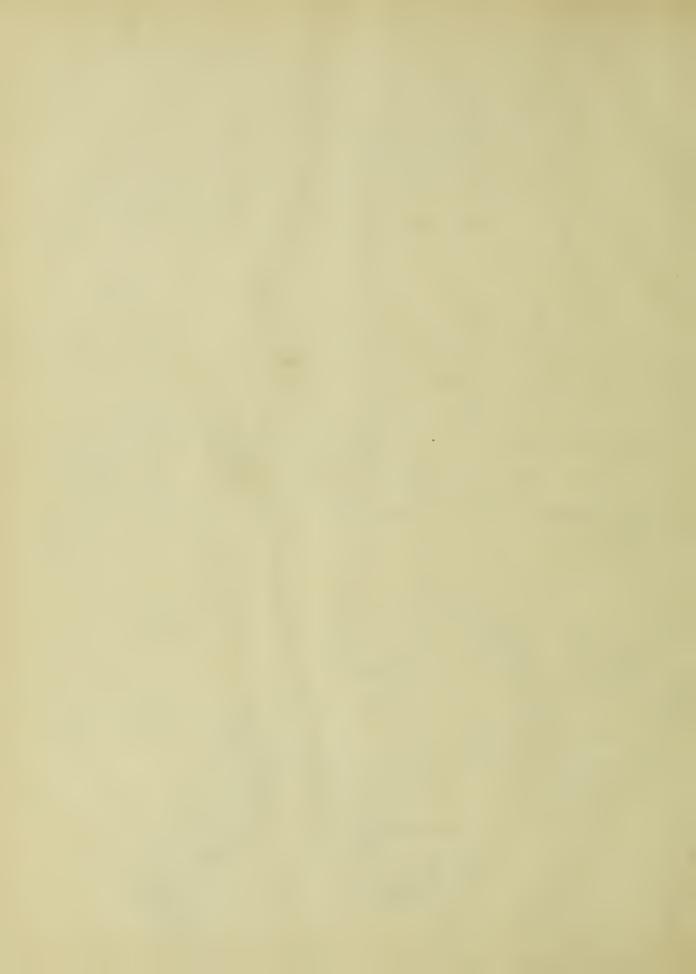
- 0.5 gm. Clay.
- 1.0 gm. Tartaric acid.
- 10.0 gm. Sodium peroxide.

The combustion was rather poor, and it seemed as though there were not a sufficient amount of heat within the bomb to carry the combustion to completeness. The amount of sodium peroxide was increased to see what the effect would be.

- 1.0 gm. Zine dust.
- 1.0 gm. Tartaric acid.
- 0.5 gm. Clay.
- 15.0 gm. Sodium peroxide.

The fusion was very poor and the reaction was quite vigorous within the bomb. Any more of an increase of sodium peroxide is liable to cause an explosion.

- 1.0 gm. Boro nitrate mixture.
- 0.5 gm. Clay.
- 10.0 gm. Sodium peroxide.
  - 0.5 gm. Starch.



This mixture did not seem to fare any better than the one before, for the combustion was very poor, in fact, much poorer than the preceding one.

- 1.0 gm. Tartaric acid.
- 0.5 gm. Clay.
- 10.0 gm. Sodium peroxide.

The fusion from the above mixture was tolerably good. It was thought that a little more starch would help matters very materially, so some was tried.

- 0.5 gm. Tartaric acid.
- 0.5 gm. Clay (accurately).
- 0.5 gm. Starch.
- 10.0 gm. Sodium peroxide.

This mixture gave a very good fusion and was taken for analysis. The silica was determined by the ordinary method of dehydration on the water bath. Duplicates, however, did not check and were somewhat low, as was the case also with the  $\text{Fe}_2\text{O}_3$  and  $\text{Al}_2\text{O}_3$ . The results for silica were 52.03% and for another experiment which was a duplicate of this with the exception that sugar was used instead of starch, 49.36%. The correct per cent of silica is 54.80%.

As a result of the last analysis, it was again concluded that the combustion was incomplete; also showing that starch works better than sugar, and it was thought that some



other oxidizing agent should be present other than sodium peroxide. The following experiments were made with that point in view.

- 0.5 gm. Clay.
- 0.5 gm. Potassium chlorate.
- 0.5 gm. Starch.
- 0.5 gm. Tartaric acid.
- 10.0 gm. Sodium peroxide.

The above substances, when ignited in the bomb, gave a rather poor combustion, and an unusually large amount of gas was generated during the fusion. The substance boiled up to the top of the bomb, making the fused mass hard to remove.

The following mixture was then tried.

- 0.25 gm. Clay.
- 0.5 gm. Potassium chlorate.
- 0.25 gm. Starch.
- 0.25 gm. Tartaric acid.
- 10.0 gm. Sodium peroxide.

This mixture gave a rather poor combustion, but the amount of gas generated was somewhat less than in the previous one. In studying the above two combustions it seemed as though a substance was needed to keep the fusion from boiling up, and at the same time develope a considerable quantity of heat. With that idea in view, the following experiments were made.



- 1.5 gm. Potassium chlorate.
- 0.5 gm. Clay.
- 0.3 gm. Naphthalene.
- 10.0 gm. Sodium perovide.

The combustion was not much better than in the previous case. However, only a small amount of gas was generated, and the reaction was very slow.

- 1.5 gm. Potassium chlorate and Boric acid.
- 0.5 gm. Starch.
- 0.3 gm. Clay.
- 10.0 gm. Sodium peroxide.

This mixture, when ignited, blew the insulation out of the bomb because the reaction was too vigorous.

- 1.0 gm. Potassium chlorate and boric acid.
- 0.3 gm. Starch.
- 0.3 gm. Clay.
- 10.0 gm. Sodium peroxide.

A very poor combustion was the result when the above substances were ignited. The ignition, or rather the reaction took place quite slowly.

- 1.0 gm. potassium chlorate and boric acid.
- 0.5 gm. Starch.
- 0.3 gm. Clay.
- 10.0 gm. Sodium peroxide.



The reaction was very vigorous, and the substance boiled to the top of the bomb. The combustion was rather poor.

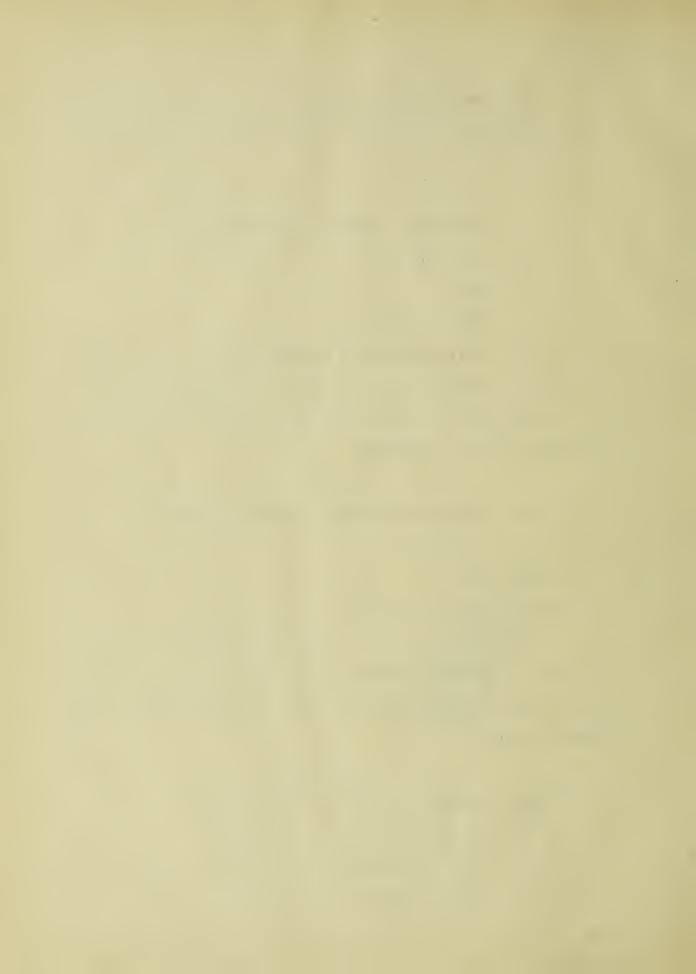
- 1.0 gm. Potassium chlorate and boric acid.
- 0.3 gm. Naphthalene.
- 0.3 gm. Clay.
- 10.0 gm. Sodium peroxide.

The above substances ignited rather slowly, but the combustion was about the best that had been obtained so far. A little more naphthalene was tried to see if the fusion could still be bettered.

- 1.5 gm. Potassium chlorate and boric acid.
- 0.4 gm. Naphthalene.
- 0.3 gm. Clay.
- 10.0 gm. Sodium peroxide.

This mixture ignited very much faster than the one above, and the reaction was considerably more vigorous, but did not boil to the top of the bomb. The combustion also, was much better.

- 1.5 gm. Potassium chlorate and boric acid.
- 0.4 gm. Naphthalene.
- 0.3 gm. Clay (accurately).
- 10.0 gm. Sodium peroxide.



A determination of the silica was made. The results were as follows:

> II. III.

Silica, Sio

54.26% 54.03% 54.62%

The duplicates seemed to agree fairly well, and also checked within the limit with the results obtained by standard methods, which is 54,80% Silica.

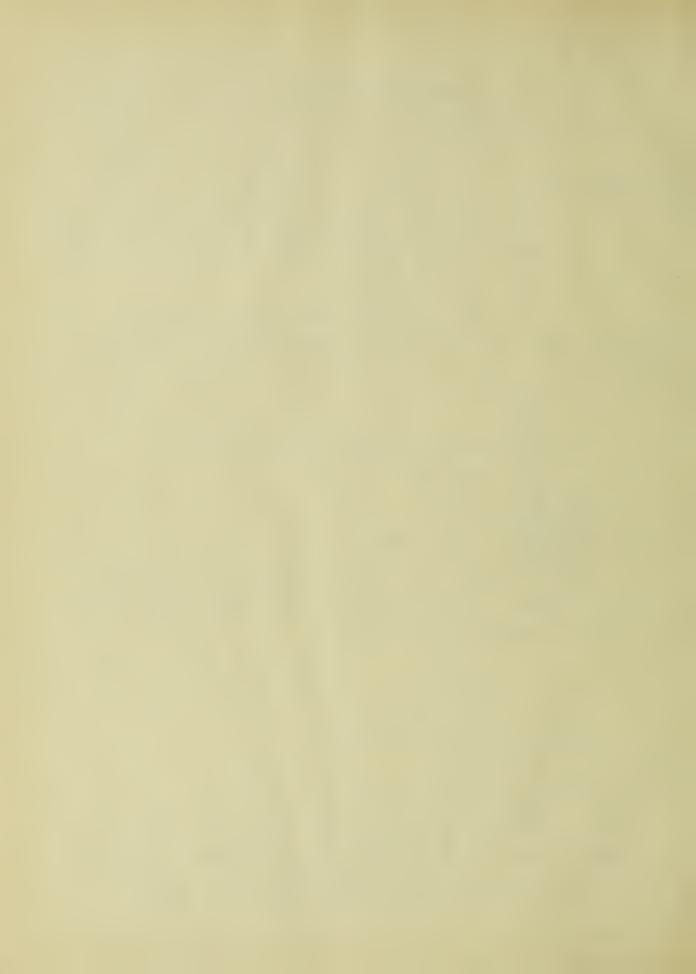
The next experiments were made on a Feldspar. The first mixute that was tried was the same as the previous one, viz.,

- 1.5 gm. Potassium chlorate and boric acid.
- 0.4 gm. Naphthalene.
- 0.3 gm. Feldspar.
- 10.0 gm. Sodium peroxide.

The result was a very poor fusion, and it ignited rather slowly. Benzoic acid was next tried and the boric acid was dropped at this point.

- 0.5 gm. Benzoic acid.
- 0.5 gm. Potassium chlorate.
- 0.3 gm. Feldspar.
- 10.0 gm. Sodium peroxide.

The combustion in this case was very good, but rather vigorous, which caused the substance to boil up to the top of the bomb, making the fusion very difficult to remove.



The purpose of the next experiment was to see in what ratio the substances used in the last mixture would ignite, give a perfect fusion, and yet not boil to the top of the bomb.

- 0.5 gm. Benzoic acid.
- 0.4 gm. Potassium chlorate.
- 0.3 gm. Feldspar.
- 10.0 gm. Sodium peroxide.

The combustion in this case was also very good, but it still boiled to the top of the bomb.

- 0.3 gm. Benzoic acid.
- 0.5 gm. Potassium chlorate.
- 0.3 gm. Feldspar.
- 10.0 gm. Sodium peroxide.

The combustion was exceptionally good and the substance did not boil to the top of the bomb. A little more benzoic acid was tried in the next experiment.

- 0.4 gm. Benzoic acid.
- 0.5 gm. Potassium chlorate.
- 0.3 gm. Feldspar.
- 10.0 gm. Sodium peroxide.

This mixture gave a very good fusion, but the substance boiled up slightly, however, causing enough difficulty



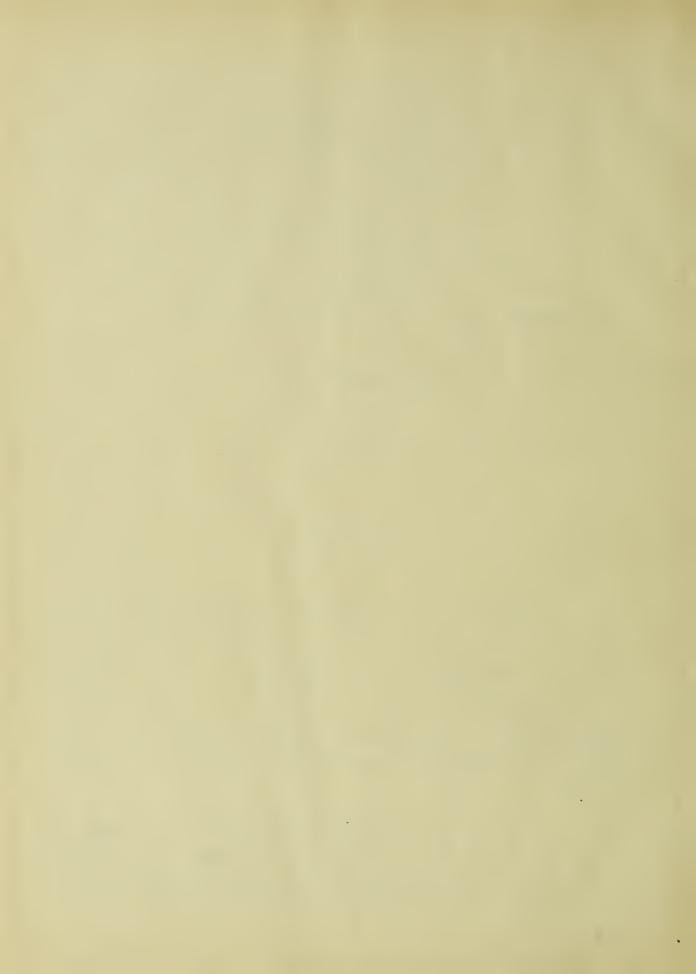
when removing the mass to not warrant the mixture for satisfactory work. The next to the last mixture seemed to give the best and most satisfactory results in all things concerned; the fusion was practically perfect; did not boil to the top of the bomb, and was very easily removed from it. The same mixture was tried on the clay and the results obtained were very similar to those obtained when the feldspar was used. This mixture contained the following substances:

- 0.3 gm. Benzoic acid.
- 0.5 gm. Potassium chlorate.
- 0.3 gm. Clay.
- 10.0 gm. Sodium peroxide.

## CONCLUSION.

Judging from the results obtained in the last analysis, it seems as though the use of sodium peroxide in a closed chamber has practically solved the question of fusion mixtures for silicates of all kinds. The figures, 54.03%, 54.26%, and 54.62% for silica using the peroxide method, agree reasonably well with the result of 54.80% for silica by a standard method.

As the silica is the most difficult constituent in silicates to get into solution, and since the results obtained by the peroxide method agree with those of standard methods,



there is no doubt in the author's mind but that the use of sodium peroxide will, in the near future, be one of the standard methods for the analysis of Feldspars and substances of a similar nature.

There is, however, considerable work to be done along this line, making further study of difficulties which may attend the estimation of the other constituents, such as iron, aluminum, etc., where large amounts of alkaline salts are present; but this circumstance is not essentially different from the conditions that exist under the usual fusion methods where sodium-potassium carbonates are used.



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